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Quantitative Analysis of Fe Oxidation State by XANES for Spinel Ferrite Nanoparticles

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Spinel ferrites are well-established materials for application due to their useful magnetic properties. Their composition, both elemental and *stoichiometric*, may be altered without major structural changes, which permits tuning their ferrimagnetic properties in a wide range. In this work, specimens containing a *non-stoichiometric* ratio of maghemite (γ-Fe_2O_3) and magnetite (Fe_3O_4) nanoparticles were studied. Fe_3O_4 is an inverse spinel with Fe^{2+}:Fe^{3+} = 1:2, and γ-Fe_2O_3 has Fe^{3+} statistically distributed in the tetrahedral and octahedral sites of a spinel lattice. It is important to correlate the relative amount of Fe_3O_4 (or γ-Fe_2O_3) in a nanoparticle specimen to the synthesis conditions used to engender the nanoparticles. It is also of interest to understand and establish a correlation between magnetization and the ratio of these ferrites through ongoing magnetic characterization experiments using a SQUID magnetometer.

These two materials have similar crystal structure and spectroscopic signature, and they cannot be differentiated with diffraction techniques due to peak broadening from nanoparticles. As there is a linear relationship between absorption edge position and oxidation state, XANES at K edge was used to determine the overall oxidation state and estimate the relative ratio of Fe_3O_4 to γ-Fe_2O_3 (Fe^{3+}) in a specimen. The edge shift as defined at half height between Fe_3O_4 and Fe_2O_3 is 1.6 eV . An example of its application to a commercial sample is shown in figure 1.

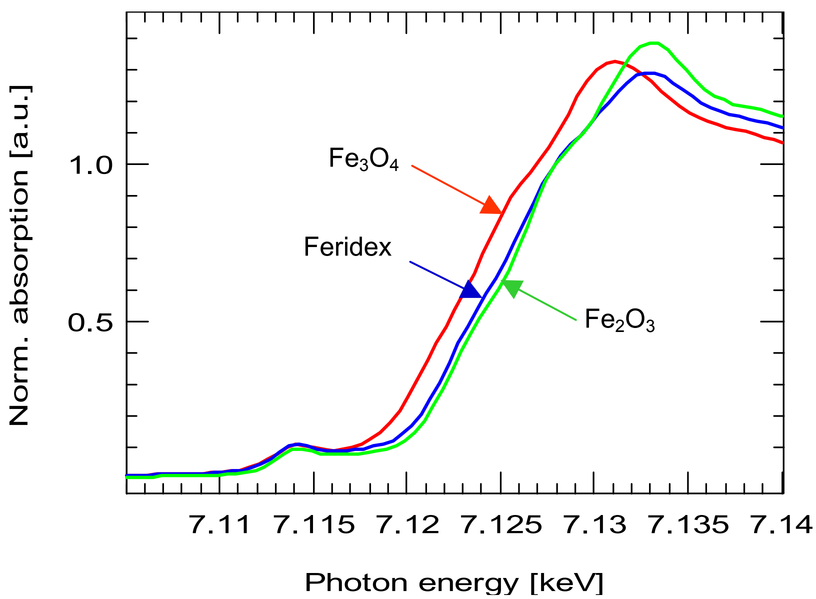

Figure 1. XANES at Fe K-edge. Feridex is a commercial ferrite nanoparticle sample containing 64% Fe_2O_3 and 36% Fe_3O_4 by weight. Its edge position clearly falls between Fe_3O_4 and Fe_2O_3 but more toward the latter, in a reasonable good agreement with nominal composition claimed by the manufacturer.